GAS ABSORPTION BY ATOMIZATION

by

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and

LT.(JG) MAVIS X. POLK, U. S. NAVY

on Assignment from

U. S. Naval Postgraduate School, Annapolis, Md.

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INTRODUCTION

Vapor - liquid transfer operations such as absorption, distillation, and stripping and the design of equipment to effect such separations have been the object of much research over the last twenty-five years. The rate of mass transfer is know to depend on such physical conditions as temperature, pressure, equilibrium relations, and upon the so called efficiencies of the particular type of apparatus.

The development of adsorption equipment began with the relatively simple process of bubbling the mixture of gases through a suitable liquid to selectively absorb one or more of the gases. It was soon recognized that the depth of the liquid and the size of gas bubbles were important factors. In other words, the design of absorption equipment moved in the direction of developing conditions of maximum surface area contact. As a matter of fact all equations developed to calculate production sizes of equipment have this rather undeterminable factor of "effective area".

It was for this reason that we had the development of the packed column and the inevitable discussions of the relative merits of the size and shape of such packing. Such columns are still in most general use and are in general built upon the experience that if built high enough, maintaining a reasonable mass velocity by control of the diameter of the column and free space in the packing and using a sufficiently high ratio of moles of absorbing liquid to moles of gas, high recoveries usually in excess of 95 per cent are realized. As

a matter of fact, six of such units in series with an overall actual height of 250 feet have been in operation in the recovery of light oils from coke oven gas.

It is obvious that the pumping costs in such units would be high but probably the greatest objection is that such systems tend to plug either by accumulation of tar like materials or by resinification of active materials on such large surfaces. This results in reduced capacity and higher pumping cost and the necessity of periodic cleaning.

These objections led to the development of spray adsorption systems starting from relatively simple sprays falling considerable distances in open or partially filled columns to the most recent design of systems in which the sprays were collected at different points in the column and recirculated in secondary spray systems at velocities in excess of the inlet rates.

At the present time great progress is being made in this design, particularly in the by-product coke industry where definite comparisons have been made with previous units. While permission has not been given to disclose the exact design, it can be said that in the recovery of ammonia from gases, one such unit has replaced eight standard saturator units and in the recovery of light oils two units in series are giving results equivalent to twelve standard units of equal height and substantially the same production. Such results lead one almost inevitably to a study of the application of atomization to absorption.

THEORY OF ATOMIZATION

The purpose of atomization of liquids is to produce a high ratio of surface to mass, resulting in an intimate contact between a gas and a liquid. While there are other so called methods of atomization such as, pressure nozzles and high speed rotating disks, the two fluid or gas stream method of atomization will be considered in this study whereby a gas stream is used to atomize a liquid through a common nozzle.

In any commercial absorption system there is obviously a certain amount of gas to be treated by an equally definite quantity of absorbing liquid, because of the closed distillation system, to recover the product. A limiting factor, therefore, must be developed whereby the gases to be treated are sufficient to atomize all of the absorbing liquid. In this sense the problem is different from that of atomization in oil burners where large excesses of air may be used.

> In effecting the atomization of a liquid . . ., force is applied to the liquid to set it in motion, and generally to constrain it to emerge at some stage of the process as an attenuated film. This film is then disrupted into shreds or filaments, either by the application of other forces or by the persisting balance of energy in the liquid derived from the initial force which promoted its flow. The shreds or filaments then break up into numbers of smaller fragments, which thereupon rapidly assume spherical form under the action of the surface tension of the liquid itself.
>
> Broadly speaking, therefore, the atomization of a liquid . . . involves enforced attenuation, followed by ligament or filament formation and disruption. The disruption of the filaments under the action of surface tension follows the πd rule, and results in the production of main droplets and Plateau droplets. Since the filaments themselves vary greatly in diameter, the resulting main droplets and the subsidiary droplets produced from them vary in size correspondingly, and in the aggregate the entire atomized spray is thus composed of droplets of a wide range of sizes.

> It must not be assumed, however, that there is a clear line of demarcation between the two phases, namely the attenuation and the filament disruption. The formation of the filaments is due in part to the applied force causing attenuation and partly to surface tension, and there is an interplay of these forces during the atomization process, perhaps also with the intervention of outside influences, as, for example, of the air directed into the spray for combustion purposes.
>
> The process of atomization will continue in this way so long as liquid is fed to the atomizer and subjected to the attenuation process; and the rate of . . . flow may be adjusted, within limits, to suit requirements without affecting the continuity of the process.

Nukiyama and Tanasawa[2-6] have done considerable work on drop size in gas stream atomization, both with convergent nozzles and sharp edge orifices. Air was used to atomize water, and the drops were collected on a specially prepared oil slide by means of a shutter arrangement. The slides were then photographed, the number of drops counted, and the mean diameter $\underline{d_o}$ calculated from,

$$d_o = \Sigma x^3 \cdot \Delta n / \Sigma x^2 \cdot \Delta n \qquad (1)$$

where $\underline{\Delta n}$ represents the number of drops, the diameters of which lie between $\underline{x}$ and $\underline{\Delta x}$. It was found that the mean diameter of the drops were uniform everywhere in the sprayed jet with the exception of the extremities.

The diameters of the water nozzles $\underline{D_w}$ were varied by these investigations from 0.2 to 1.0 mm and those of the air nozzles $\underline{D_a}$ from 1 to 5 mm. The air velocity $\underline{v_a}$, water velocity $\underline{v_w}$, and air - water volume ratio $\underline{Q_a/Q_w}$ were also varied, and it was found that the drop size decreased with increasing air velocity for both types of nozzles. It was also found that it is the ratio $\underline{Q_a/Q_w}$ that effects the mean diameter $\underline{d_o}$ and

not the values of v_w, D_w, nor the nature of the flow of water, when the relative velocity ($v_a - v_w$) was kept constant. The mean diameter decreases with the increase of Q_a/Q_w up to approximately 5000, but beyond that the mean diameters are constant for a given air velocity. For values Q_a/Q_w beyond 5000, it was found that the mean diameter decreased with an increase in relative velocity, and that the results could be represented by the formula

$$d_o = 5300/v \qquad (2)$$

for the convergent nozzle and by

$$d_o = 3400/v \qquad (3)$$

for the sharp edge orifice, where d_o is the mean diameter in microns, and v is the relative velocity in meters per second. These values are for water only, and vary with the liquid to be atomized. The results show apparently that the sharp edged orifice is superior to the convergent nozzle in the atomizing action. The relative velocity v and the ratio Q_a/Q_w were kept constant and the size of the air and water nozzles were varied, but no obvious difference in drop size was noted.

These investigators also proposed two empirical equations, one predicting the surface average drop size and the other on particle size distribution by atomization with high velocity gas streams.

> In the present paper, it is found that the distribution of drops at an arbitrary point in a sprayed jet can be expressed by a simple empirical formula of the type
>
> $$dn = ax^p e^{-bx^q} \cdot dx$$

where a, b, p, and q are constants, and x is the mean diameter of dn drops restricted to the limiting diameters $(x - dx/2)$ and $(x + dx/2)$; i.e. within the range of diameters dx.

When the velocity and the quantity of the air stream are both high and sufficient (v_a = 150 to 300 m/s), $Q_a/Q_1 > 5000$, we get the values of p and q as 2 and 1 . . . . Thus we obtain

$$dn = 0.5\, nb^3x^2\, e^{-bx} \cdot dx$$

where n is the total number of drops appeared in a certain photomicrogram . . . . If the velocity and the quantity of the air streams are not both high and sufficient, q becomes less than 1 and the degree of uniformity decreases.[4]

The following expression is derived for the mean diameter of the drops

$$d_o = 585 \frac{\sqrt{\sigma}}{v\sqrt{\rho}} + 597 \left(\frac{\mu}{\sqrt{\sigma\rho}}\right)^{0.45} \left(1000 \frac{Q_1}{Q_a}\right)^{1.5}$$

where d_o (measured in microns) = the diameter of a single drop having the same volume - surface ratio as the total sum of the drops, v m/s = the relative velocity between the liquid and air stream ρ g/cm³ = the density of the liquid, σ dyne/cm = the surface tension of the liquid, μ dynes/cm² = the viscosity of the liquid, Q_a cm³ = the quantity of the air; Q_1 cm³/s = the quantity of the liquid.

From the above expression it may be concluded that the magnitude of the drops is mainly governed by the value of $\sqrt{\sigma}/v\sqrt{\rho}$, when the ratio Q_a/Q_1 is large, whereas the viscosity is of minor importance . . . . But when the ratio Q_a/Q_1 becomes less, the magnitude is mainly governed by the value of $(\mu/\sqrt{\sigma\rho})^{0.45}$ $(1000\, Q_a/Q_1)^{1.5}$, and the surface tension gives only a slight influence[5] . . . .

These equations were studied and verified by Lewis et al[7] and also by Mugele and Evans.[8]

EXPERIMENTAL APPARATUS

The apparatus used in this experiment is shown diagrammatically in Figure 1. The carbon dioxide supply was a fifty pound cylinder of carbon dioxide. The carbon dioxide rate was controlled by means of a standard carbon dioxide reducing valve, and the quantity was measured by means of a gas rotameter. The cooling effect due to the throttling of the carbon dioxide caused fluctuation in the rate of flow.[9] These fluctuations were eliminated by allowing the carbon dioxide to flow long enough to maintain essentially constant temperature and hence constant flow rate through the throttling valve before taking readings. The air was supplied by an Ingersoll Rand air compressor. The air rate was maintained constant by means of a needle throttling valve, and the rate was measured by means of a second gas rotameter. The carbon dioxide and air lines were joined upon leaving the respective rotameter and the mixture was led into the mixing chamber of the nozzle Figure 2.

The water supply was taken from the city water main. It was put through a glass wool filter which was placed in the supply line. The water rate was measured by a liquid rotameter and controlled by means of two control valves, one placed before and one after the rotameter in the line. The water line contained a bleed off valve for purging. The water was introduced into the nozzle from the back Figure 2 and then mixed with the gas mixture in the mixing chamber and then discharged into the collector as a fog.

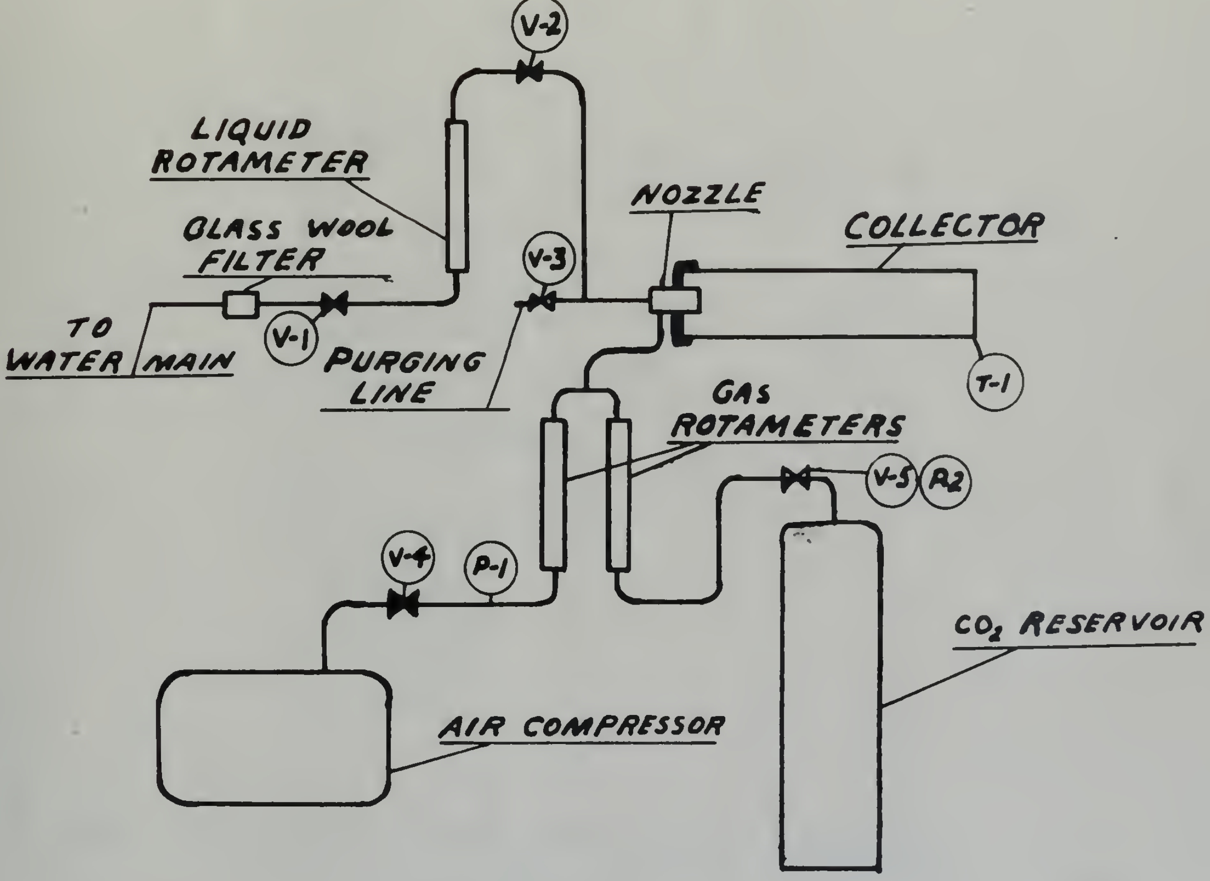
The collector was sealed to the nozzle at the nozzle end and open at the discharge end. Several different types and sizes of collectors were used as listed in tables # 3 & 4.

The atomizing nozzle used was a sharp-edged gas atomizing nozzle, Figure 2. The design was a modification of that used by Nukiyama and Tanasawa.[4] The water orifice was 0.013 inches and the outlet or gas - water mixture nozzle was 0.150 inches in diameter.

V_1, V_2, V_3, V_4, V_5 - CONTROL VALVES

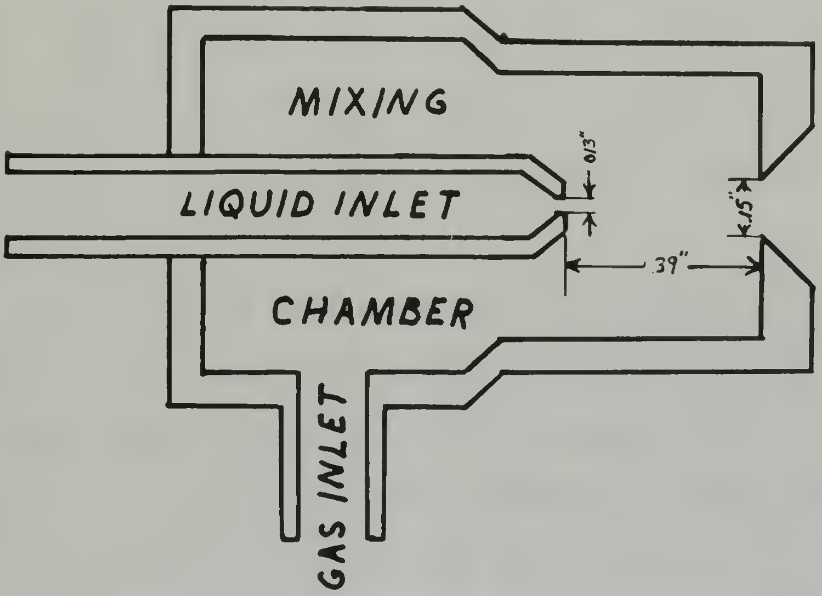
P_1, P_2 - PRESSURE POINTS

T_1 - TEMPERATURE POINT



DIAGRAMATIC SKETCH OF APPARATUS

FIGURE 1



GAS LIQUID ATOMIZATION NOZZLE
FIGURE 2

EXPERIMENTAL PROCEDURE

Metered streams of carbon dioxide and air were mixed and fed into the atomizing nozzle. Rates of both streams were maintained constant by manually operated throttling valves. The ratio were observed by means of gas rotameter readings in each of the lines. The rotameters were placed so that the readings were obtained just before mixing of the gases.

Tap water from the Bethlehem City water main was used. The water was introduced to the nozzle through a liquid rotameter. The temperature of the water was that of the under ground lines and therefore varied somewhat from day to day. The water rate was maintained constant manually by means of a control valve.

The water entered the atomizing nozzle from the back and then into the atomizing chamber through a 0.013 inch orifice. The gas mixture entered the atomizing chamber from the side, picked up the water and the mixture was then dispersed into the collector through a 0.150 inch orifice as a fog. This fog was discharged into a collector. The collector consisted of a glass pipe. Pipes of various lengths and diameters were tried so as to note the effect that each had on the fog pattern. The end of the tube through which the nozzle fitted was sealed around the nozzle to prevent air from being drawn in by the fog leaving the nozzle. The discharge end of the collector was left open and the samples for analysis were collected at the discharge end. The temperature of the water carbon dioxide mixture was then taken at the discharge end of the collector.

Runs were made in the following manner: The inlet gas composition and the gas and liquid rates were fixed. They were allowed to run until steady rates could be maintained. This was necessary due to the cooling effect of the carbon dioxide through the reducing valve which would cause fluctuations in the carbon dioxide rate for awhile after starting. After rates became steady liquid samples were taken from the end of the collector and analyzed. The temperature of the water - carbon dioxide mixture was taken at the sampling end of the collector.

METHOD OF ANALYSIS

There were two methods of analysis of the water - carbon dioxide solution used. In the first method 10 ml samples were collected in a pipette and introduced into a flask. The H_2CO_3 was neutralized by titrating with 0.0527 N $Ba(OH)_2$ directly. Runs 1 - 37 were analyzed by this method.

It was felt that some of the carbon dioxide was being lost to the surrounding air during sampling and titration so a second method of analysis was tried.[9] In this method the sample was collected as before in a 10 ml pipette, but was introduced into a flask containing 10 ml of 0.0527 N $Ba(OH)_2$. The end of the pipette was placed beneath the surface of the $Ba(OH)_2$ when transferring the sample. The excess $Ba(OH)_2$ was then neutralized by titrating with 0.0935 N. HCl. Runs 38 - 48 were made to check these two methods of analysis. It was apparent that carbon dioxide had been escaping in the first method of analysis so the second method was adopted for the remaining experimental work.

TABLE 1

Determination of time necessary to obtain constant sample using iron pipe 10'11"x2 1/2" as collector.

Run	Time min.	Temp. °F	Water Rate min.	CO_2 Rate C.F.M.	CO_2 absorbed gr. CO_2 per 100 grs H_2O
1	1	66	93	2.26	0.056
1	2	66	93	2.26	0.088
1	3	66	93	2.26	0.091
1	4	66	93	2.26	0.163
1	5	66	93	2.26	0.165
2	1	69	81	2.29	0.086
2	2	69	81	2.29	0.094
2	3	69	81	2.29	0.144
2	4	69	81	2.29	0.137
2	5	69	81	2.29	0.146

TABLE 2

Effect of variation of CO_2 rate. Collector same as Table 1.

Run	Temp. °F	Water Rate cc/min	CO_2 Rate C.F.M.	CO_2 absorbed gr. CO_2 per 100 grs H_2O
3	80	96	0.42	0.076
4	80	96	0.56	0.098
5	80	96	1.05	0.117
6	80	96	1.52	0.136

TABLE 3

Effect of variation of gas composition. Temperature was 70°F. Collector was same as in Table 1.

Run	Press psia	Water Rate cc/min	Air Rate C.F.M.	CO_2 Rate C.F.M.	%CO_2 inlet	CO_2 absorbed gr. CO_2 per 100 grs H_2O
7	34.7	128.0	3.35	0.85	20.3	0.012
8	24.7	128.0	1.90	0.76	28.6	0.026
9	22.7	128.0	1.57	0.73	31.7	0.032
10	23.7	125.5	1.37	0.94	40.6	0.056
11	29.7	125.0	1.30	1.30	50.0	0.092

TABLE 4

Effect of varying size of collector.
CO_2 rate 1.26 C.F.M. air rate 2 C.F.M.
Pressure 32.7 psia. 38.6% CO_2 in inlet gas

Run	Type Collector	Water Rate C.F.M.	Temp. °F	CO_2 absorbed gr. CO_2 per 100 grs H_2O
12	8'x2 3/4" pipe horiz.	92	69	0.084
13	4'x2 3/4" pipe horiz.	92	69	0.129
14	No pipe	92	69	0.083
15	4'x2 3/4" pipe vert.	67	69	0.038
16	4'x2 3/4" pipe vert.	67	69	0.047
17	4'x2 3/4" pipe vert.	85	70	0.035
18	4'x2 3/4" pipe vert.	85	70	0.035
19	2"x1/2" rubber hose	90	70	0.058
20	4'x2 3/4" pipe horiz.	88	70	0.110
21	3'x5 1/2" pipe horiz.	88	70	0.026
22	3'x5 1/2" pipe horiz.	88	70	0.031
23	1'x1 7/8" pipe horiz.	90	70	0.038
24	1'x1 7/8" pipe horiz.	90	70	0.050
25	2'x7/8" pipe horiz.	90	70	0.045
26	2'x7/8" pipe horiz.	94	70	0.033
27	2'x7/8" pipe horiz.	90	70	0.031
28	2'x7/8" pipe horiz.	90	70	0.022
29	2'x7/8" pipe horiz.	82	70	0.040
30	2'x7/8" pipe horiz.	85	70	0.043
31	2'x7/8" pipe horiz.	85	70	0.041
32	2'x7/8" pipe horiz.	92	70	0.028
33	2'x7/8" pipe horiz.	96	70	0.043
34	1'x2 3/4" pipe horiz. (constricted end)	95	70	0.037
35	1'x2 3/4" pipe horiz. (constricted end)	85	70	0.035
36	1'x2 3/4" pipe horiz. (open end)	92	70	0.031

TABLE 5

Comparision of titration methods. Gas rate and composition, pressure and temperature were held constant. Water rate was 92 cc/min. for runs 37 & 38 and 85 cc/min. for runs 39 - 47. Collector was a 4' x 2 3/4" pipe in a horizontal position.

Run	Gr. CO_2 absorbed per 100 grs H_2O. Analysed by titration with $Ba(OH)_2$	Gr. CO_2 absorbed per 100 grs H_2O. Analysed by placing 10 ml of sample in 10 ml of $Ba(OH)_2$ and titrating excess $Ba(OH)_2$ with HCL
37	0.025	0.047
38	0.027	0.057
39	0.037	0.050
40	0.038	0.060
41	0.035	0.060
42	0.028	0.044
43	0.037	0.059
44	0.035	0.059
45	0.033	0.054
46	0.037	0.058
47	0.038	0.060

TABLE 6

Effect of variation of gas composition. Water rate was constant at 100 cc/min. Collector same as in Table 5.

Run	Temp. °F	Press psia	% CO_2 inlet	Air Rate C.F.M.	CO_2 Rate C.F.M.	CO_2 absorbed gr. CO_2 per 100 grs H_2O
48	70	23.0	32.0	1.55	0.73	0.050
49	70	29.7	28.3	2.10	0.83	0.040
50	68	31.7	16.3	3.08	0.60	0.030
51	76	29.7	50.0	1.20	1.20	0.073
52	76	23.0	32.9	1.54	0.74	0.054
53	76	29.7	27.9	2.07	0.84	0.038

EXPLANATION OF TABLES

Air rates and carbon dioxide rates in all tables have been corrected to cubic feet per minute at one atmosphere pressure and 70°F. The pressures listed in the tables are of the metered gas and not that of the gas - water mixture. The pressure of the gas - water mixture was one atmosphere.

Table 1 was prepared to show the time necessary for a representative sample to appear at the sampling end of a pipe 11' 10" long. Table 2 indicates the effect of varying gas rate upon atomization and hence the amount of gas absorbed from pure carbon dioxide. Table 3 shows the results of using a carbon dioxide - air mixture for the gas and varying the rates and composition of the gas mixture. Table 4 shows the effect of different type collectors on the amount of carbon dioxide absorbed. The carbon dioxide and air rates were maintained constant but the water rate varied somewhat due to the water main pressure. Runs 12 - 16 were made one day and runs 17 - 36 were made the next day. Table 5 shows a comparison of analytical methods. Two samples were taken on each run. Runs 37 and 38 were made one day and runs 39 - 47 were made the next day. All samples of runs 1 - 36 had been analyzed by titration with barium hydroxide. Table 6 indicates the effect of changing gas composition on the amount of carbon dioxide absorbed. The method of analysis used on the samples for table 6 was that of neutralization of the sample with excess barium hydroxide and titration of the excess alkali with hydrochloric acid.

COMPARISON OF RESULTS

The results of this experiment were compared with results obtained from a packed column experiment[10] in order to judge the effectiveness of atomization in adsorption. The data from the literature[10] was converted to grams of carbon dioxide per hundred grams of water as a basis of comparison. Three runs from Table II, page 991, reference 10 were found to have percentage composition nearly the same as runs made in the present experiment and these runs were selected for comparison.

These comparisons can be used only as rough indications because of inability at that early date (1927) to construct columns large enough and because of the lack of modern instrumentation.

Sample calculations using data from reference 10 to convert results to grams of carbon dioxide per 100 grams of water.

Data taken from Table #II, page 991, reference 10

16.0 = % of CO_2 in inlet gas
21.4°C = Temperature of inlet gas
3.94 lit/min = inlet gas rate
7.2 = % of CO_2 in exit gas
22.4°C = Temperature of exit gas
3.56 lit/min = exit gas rate
7.6 lit/min = water rate
8.8°C = water temperature

$3.94 \times .16 \times \frac{273}{294.4}$ = .584 liters of CO_2 in inlet gas con to S. C.

$3.56 \times .072 \times \frac{273}{295.4}$ = .237 liters of CO_2 in exit gas con to S. C.

$\frac{(.584 - .237)}{22.4 \times 76} \times 44 = .009$ grams of CO_2/100 grams of H_2O

TABLE 7

Method of adsorption	Composition % CO_2	CO_2 absorbed gr. CO_2 per 100 grs. H_2O
Packed Column	16.0	0.009
Atomization	16.3	0.029
Packed Column	29.2	0.018
Atomization	27.9	0.037
Packed Column	33.1	0.021
Atomization	32.1	0.055

CONCLUSIONS

The work done in this experiment was of an exploratory nature into the field of adsorption by atomization. The comparision of results with those found in the literature on adsorption by use of packed columns shows that apparently a higher degree of absorbate in absorbent can be obtained by the method of atomization then by the method of packed columns.

When pure carbon dioxide was used as the gas, it was found, Table 1, that the handbook" equilibrum values of carbon dioxide in water could be attained almost instantaneously by the method of atomization.

It should be pointed out that the experimental equipment used was of a fairly rough design and also that the accuracy of the data from the literature which was used as a basis of comparision was not verified by the authors. The authors contemplate doing further work in this field using better equipment and different systems.

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